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PATENT SPECIFICATION

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COMPLETE SPECIFICATION.

Detergent Tablets and Processes for Manufacturing Them.

We, COLGATE-PALMOLIVE COMPANY, a Corporation organised and existing under the Laws of the State of Delaware, United States of America, of 300 Park Avenue, 5 New York 22, New York, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us and the method by which it is to be performed, to be particularly described 10 in and by the following statement:—

The present invention relates to a detergent tablet which is resistant to abrasion and accidental breakage when dry and is also adequately disintegrable in water in normal 15 laundering. The invention also relates to a process for manufacturing such tablets.

According to one aspect of the invention, 20 a process for manufacturing a briquetted detergent tablet comprises mixing together water-soluble non-ionic organic detergent and a normally solid water-soluble solid inorganic salt, producing therefrom a particulate detergent containing up to 21% moisture, applying an aqueous solution of 25 water-soluble silicate, preferably by spraying, to coat the particles with 3 to 20% soluble silicate and to increase the moisture content thereof to 16 to 25%, pressing the 30 detergent particles into a solid form-retaining briquetted tablet at a pressure low enough to form a tablet which is readily disintegrable in water and applying to the tablet 0.25 to 5% of a readily water-soluble organic 35 film-forming polymer to form on the briquette surface a water-soluble film which is of strength sufficient to help make the detergent tablet resistant to abrasion and accidental breakage, when dry, and of a solubility such that the detergent tablet is readily 40 disintegrable in water.

Between the steps of coating the particles [Price -

with soluble silicate and pressing them into briquetted tablet form, there is preferably added to the detergent particles 0.5 to 5% of finely divided water-insoluble inorganic silicate to form another coating, held to the particles by the water-soluble silicate. The coating with insoluble silicate is a preferred feature of the invention because it measurably improves the ease of pressing the detergent particles into tabletted form and permits high speed production on a commercial scale.

According to another aspect of the invention, 55 a water-soluble detergent briquette which is resistant to abrasion and accidental breakage when dry, and disintegrates readily in water, comprises a multiplicity of solid particles composed of a homogeneous mixture of water-soluble non-ionic organic detergent and water-soluble inorganic builder salt, of 16 to 25% moisture content, in the form of adhering particles coated with 3 to 20% water-soluble silicate, the coated particles being lightly compacted and fused together to form a shaped briquette, which is coated with an adhering and binding film of 0.25 to 5% of a readily water-soluble film-forming polymer.

Following production of the briquettes, 70 they may be packed in polyethylene bags or similar substantially moisture-tight containers to reduce moisture loss from the tablets.

In performing the process, it is preferred 75 to mix together the major constituents of a heavy duty synthetic organic detergent composition, including non-ionic detergent and inorganic builder salts, in an aqueous solution or dispersion, usually containing 20 to 70% solids. The uniform slurry is atomized and sprayed into a heated drying gas to form 80 a spray dried detergent in particulate form,

preferably of a "low-sudsing" type formula. Instead of spray drying, other production techniques may be employed to produce such detergent powders.

- 5 The dry or partially dried detergent is agitated, as by tumbling in a rotating inclined drum, and while being so moved, is sprayed with a solution of water-soluble silicate. The silicate is spread over the surfaces of the particles by the tumbling action and causes the finer particles present to adhere to others to form particles of larger size. This agglomeration is often accompanied by a decrease in density. In addition to altering product density, the silicate conditions the particles so that they will adhere together better in subsequent briquetting operations.
- 10 After coating with silicate and continuing mixing for about one to five minutes, a finely divided water-insoluble silicate powder, preferably talc that will pass a 200 mesh sieve (0.074 mm. opening), may be distributed over the particles previously coated with soluble silicate. The talc coating helps to arrest agglomeration and improves ease of briquetting. The talc is preferably applied in the same mixing drum as the silicate, preferably as a fluidized powder blown into the mixer by air pressure. After about 5 to 15 minutes mixing, the treated detergent particles are withdrawn, over-sized agglomerates are screened out and the product, preferably as agglomerates of particle size within the range of 6 to 60 mesh (3.36 to 0.250 mm. openings), is removed. The coating process described above may be either of the batch type or continuous, the latter being preferred for high speed commercial procedures. Sieve sizes given are of the United States Sieve Series, which corresponds closely to the Tyler Standard Screen Scale Sieves.
- 15 The coated detergent may be immediately pressed into briquetted tablet form or may be temporarily stored for several hours before compacting. The free flowing particles are fed to mould cavities and are lightly compacted to desired shape, usually at a pressure in the range 10 to 100 pounds per square inch, preferably 40 to 90 pounds per square inch. The pressure employed is that at which the tablets are made resistant to abrasion and accidental breakage, and are still readily disintegrable in water. Thus, in a simulated washing machine test, freshly made tablets, also coated with organic polymer as described below, should disintegrate in agitated water at room temperature in less than 90 seconds, preferably less than 60 seconds. When aged, the hardened tablets may take as long as 4 minutes to break up completely. Although it is desirable to have the most rapid solubility characteristics possible, and for this reason the finished

tablets are preferably packed in sealed containers, even the aged tablets not so packed still wash satisfactorily.

The lightly compacted tablets are next sprayed with a protective coating of an organic film-forming polymer, preferably polyvinyl alcohol, usually as an aqueous solution applied in spray form. The entire briquette surface is covered and a strong bond between the polyvinyl alcohol and the soluble silicate coating is obtained. To speed production and make a hard protective coating on the detergent tablet, the polyvinyl alcohol coating is dried by infra-red heating lamps. The finished tablets are then packed in substantially moisture-tight containers, e.g. plastic bags or sleeves, boxed, cased and shipped. The plastic bags are preferably of thin film material, such as polyethylene, and are sealed.

The materials of the present compositions and the processing operations will now be described in greater detail.

The water-soluble non-ionic organic detergent is preferably a lower alkyl phenoxy poly lower alkenoxy alkanol. In this connexion the term "lower" means that the group concerned contains not more than 8 carbon atoms. Nonyl phenoxy polyethenoxy ethanol of 9 to 10 ethenoxy groups is a specific example of this group found to be excellent. In the preferred class of compounds mentioned the alkyl group is usually of 6 to 14 carbon atoms and the alkenoxy chain is of 4 to 12 units long, each unit comprising from two to four carbon atoms. Commercial products of this type are usually mixtures, and mixes of this and other types of water-soluble non-ionic detergents may be employed. Thus, other non-ionics, such as the block copolymers of ethylene oxide and propylene oxide (Pluronics), reaction products of higher fatty alcohols and lower alkylene oxide (Emulgophenes), polyoxyethylenated higher fatty acids, polyethenoxy and poly lower alkenoxy esters and ethers of sugar alcohols, especially of sorbitol and mannitol, may be used in addition to the alkyl phenoxy polyalkenoxy alkanol and in some cases in replacement thereof. In the present detergent compositions the proportion of non-ionic detergent, on a final product weight basis, as are all other proportions given unless otherwise indicated, is usually 4 to 14%, preferably 7 to 12%.

In addition to non-ionic detergent, an anionic detergent may also be present, usually primarily to produce some foam and to contribute its cleaning power to the composition. A preferred detergent of this type is sodium tridecyl benzene sulphonate, a mixture of detergents the alkyl groups of which are derived from a mixture of propylene tetramer and pentamer. The alkyl groups may be of other structures of 10 to 18 car-

- bon atoms, preferably 12 to 15 carbon atoms, and these compounds may be used as their other water-soluble metal salts of similar properties. In place of some or all 5 of the alkyl benzene sulphonate, one may employ other anionic sulphated or sulphonated organic detergent compounds containing fatty alkyl or acyl groups of 10 to 18 carbon atoms, preferably 12 to 15 carbon 10 atoms, such as sodium lauryl sulphate, sodium coconut oil fatty acids monoglyceride sulphate and sodium N-higher fatty acyl-N-methyl tauride. The proportion of anionic detergent should normally be from 1 to 5%. 15 A foam stabilizer, such as a long chain fatty compound having an alkyl group of 12 to 18 carbon atoms, preferably a mixture of cetyl and stearyl alcohols, may also be included to thicken the foam produced by the anionic detergent. Ordinarily, 0.5 to 5% and preferably 0.5 to 3% of this material is enough to accomplish that purpose. 20 The inorganic water-soluble normally solid salt is preferably a builder salt of the polyphosphate type, such as pentasodium tripolyphosphate. Other useful builders and fillers include alkali metal sulphates, sodium and potassium carbonates, tetrasodium pyrophosphate and sodium silicates of various 25 $\text{Na}_2\text{O}/\text{SiO}_2$ ratios. These give the tablets a useful and functional base for the organic materials. From 30 to 75% of the product may be soluble inorganic salt, and preferably 30 to 50% sodium tripolyphosphate is 30 used in mixture with smaller proportions of sodium silicate ($\text{Na}_2\text{O}/\text{SiO}_2$ ratio of approximately 1 : 2.35) and sodium sulphate. 35 Using the spray drying method mentioned earlier, a slurry of the above materials in an aqueous medium is dried to a moisture content of 7 to 14%. By this and other methods, moistures of up to 21%, usually 40 2 to 18%, are obtainable and particles of such moisture contents can be made into 45 satisfactory tablets by the present methods. If the spray dried particles are high in fines or coarse pieces, they should be screened or classified to substantially all (90% or more) held by a 100 mesh sieve (0.149 mm. opening) with none on 6 or 8 mesh (2.38 mm. opening). 50 The silicate solution sprayed onto the detergent particles when they are being tumbled or otherwise agitated is a water-soluble alkali metal silicate. Of the alkali metal silicates, the best is a sodium silicate of an $\text{Na}_2\text{O}/\text{SiO}_2$ ratio from 1 : 2 to 1 : 3, most preferably approximately 1 : 2.35. This 55 silicate is made into an aqueous solution which may be of 25 to 46% solids content. Generally the silicate solution will be of 35 to 43.5% solids content, preferably 35 to 37%. The selected concentrations given 60 can be satisfactorily stored and sprayed. The proportion of water solution of sodium sili- 65 cate sprayed onto the moving detergent particle surfaces is usually 15 to 30%, preferably about 20%. Thus, 15 to 30 parts solution are sprayed onto 85 to 70 parts of detergent particles, neglecting a minor correction to compensate for the small amounts of coating applied later. The 3 to 15% silicate solids coating added, preferably 6.5 to 70 8.5%, helps to agglomerate the particles to a 6 to 60 mesh particle size range (about 0.25 to 4 millimetres diameter) and helps to create a strong briquette. It also raises moisture content to 16 to 25%, 17 to 24% and preferably 17.5 to 20.5%, the best concentrations for the briquetting operations. 75 After coating with the silicate and continued mixing and agglomerating, the tumbling particles may next be coated with a suitable very finely divided talc. Other magnesium silicates or other suitable finely divided water-insoluble inorganic silicates, such as kaolin, feldspars or micas, of equivalent properties may be used with or 80 in place of talc. 0.5 to 5% talc is sufficient to hinder further agglomeration and improves the speed and ease of pressing. After the particles are coated with talc, they are removed from the tumbling zone and are immediately ready for pressing into tablets. 85 In pressing, the detergent particles are 90 flowed into moulds or pockets in high speed shaping equipment. Opposed dies of polytetrafluoroethylene in a chrome plated metal sleeve or die-box such as one coated with silicone wax, compact the particles into flat, circular tablet form, bevelled and with a diametric score line to facilitate breaking in half, if desired. The dies are set with stops to make tablets of constant thickness. The same volumes of detergent are charged 95 accurately so that compacting pressures may be kept within the correct range of 10 to 100 pounds per square inch. After compacting and removal from dies, the pressed tablets, due to their silicate coatings, can 100 be handled without fear of objectionable breakage. However, such tablets would not be permanently satisfactory unless subsequently coated with a water-soluble polymeric film-forming compound, such as polyvinyl alcohol. 105 110 115 The polyvinyl alcohols of commerce usually contain minor proportions of a lower fatty acid ester, polyvinyl acetate. This may be present in polyvinyl alcohols employed in carrying out the processes of this invention to the extent of about 10 to 30%. The useful polyvinyl alcohols are also of a weight degree of polymerization between 120 30 and 300. A good polyvinyl alcohol for 125 use in accordance with this invention has a weight average degree of polymerization of 50 to 250 (viscosity of 2 to 6 centipoises) and a polyvinyl acetate content of 15 to 25%, and a most preferred material has a 130

weight average degree of polymerization of about 100 (viscosity of 3.5 centipoises) and polyvinyl alcohol content of about 80% with about 20% polyvinyl acetate.

5 The polyvinyl alcohol should contain relatively little insoluble matter, should be colourless, odour-free and of neutral pH. Deviations from the above requirements may be made where warranted but, in general, a polyvinyl alcohol satisfying these requirements would be most acceptable for household detergent products. Instead of the described polyvinyl alcohol, other readily water-soluble synthetic organic film-forming polymers of similarly useful properties can be used, but usually such compounds will not be as effective. However, polyvinylpyrrolidone, sodium carboxymethyl cellulose, hydroxypropyl methyl cellulose and similar 15 polymeric substances can produce water-soluble films or coatings for briquette surfaces. When used, it is preferred to mix them with polyvinyl alcohol so that the special advantages attending the presence of 20 that material may be retained.

Coating of the detergent tablet is preferably done by spraying onto the surfaces a liquid comprising the polyvinyl alcohol dissolved in water. The aqueous solution 25 should be sprayed or atomized in fine droplet form as evenly as possible onto the surface of the tablet. The spray solution may contain glycerol or other lower polyols to serve as plasticizers to help to keep the polyvinyl alcohol flexible and resilient, detergents or wetting agents to improve the wetting of the briquetted tablets, and neutralizing agents, e.g. sodium bicarbonate, to improve the odour of the polyvinyl alcohol by 30 reacting with free acid that might be present. Among other plasticizers that could be used are glycols, e.g., ethylene glycol and sugar alcohols, such as sorbitol.

The amount of polyvinyl alcohol sprayed 35 onto the tablet surface should be from 0.25 to 5% of the briquette weight, preferably 0.25 to 1%. The polyvinyl alcohol is preferably applied as a 10 to 25% solution, such as a 17% solution in water, the proportion of such solutions being used being 40 from 1 to 10%, preferably about 6% of the, tablet weight. When sprayed onto the detergent briquette surface, the polyvinyl alcohol and its solvent fill the voids between 45 particles and also cover the surfaces of the particles. Because the briquette before coating is not perfectly smooth, it is evident that the coating will be thicker in some spots than in others but it may be said that the 50 average thickness will be approximately from 0.01 to 0.8 millimetre, preferably 0.02 to 0.1 millimetre.

After coating with polyvinyl alcohol solution, the tablet may be surface dried by 55 forced air, heated air, infra-red rays or other

suitable drying means to remove essentially all the solvent accompanying the polyvinyl alcohol in the coating spray. Thus, the final product will be of about the same moisture content as that obtained in the particles before pressing. After drying, the tablets may be packed immediately into cartons ready for shipment and use. It is usually preferred to have moisture barriers in such cartons to assist in maintaining the correct moisture content in the briquette during what may be an extended storage period. Briquettes made in accordance with this invention may be shipped commercially without breaking and even though stored for months before use will still disintegrate and dissolve rapidly enough when added to the tub of any of the conventional washing machines. They will also pass severe strength and solubility tests set for such products, withstanding a drop of at least one foot onto a hard surface, e.g. a metal plate, without breaking, and also disintegrating in agitated water at 100° Fahrenheit in a washing machine within a period of no greater than 4 minutes, usually within one minute. The washing machine referred to is a commercial top-loading machine with a centre post agitator of average operating and design characteristics.

The following examples illustrate the invention. All percentages and proportions in the examples and elsewhere in this specification are by weight unless otherwise indicated.

<i>Example 1</i>			
Higher alkyl phenoxy polyethenoxy ethanol (alkyl group of about 9 carbon atoms and ethenoxy chain of 9 to 10 units)	11.8	105
Sodium higher alkyl benzene sulphonate (alkyl group of 12 to 15 carbon atoms)	4.7	
Higher fatty alcohol (mixture of cetyl and stearyl alcohols)	0.9	110
Sodium phosphates (resulting from spray drying pentasodium tripolyphosphate)	41.2	
Sodium sulphate	28.3	
Sodium silicate ($\text{Na}_2\text{O}/\text{SiO}_2 = .43$)	...	3.5	115
Adjuvants (brightener, anti-redeposition agent, preservative, anti oxidant, perfume)	0.9	
Moisture	8.7	
		100.0	120

This formula was obtained by spray drying an aqueous slurry by atomization into heated drying gas, according to normal spray tower procedures. The spray dried detergent obtained was screened and was substantially all (over 90%) within the 8 to 100 mesh United States Standard Sieve Series range. Particles outside this range

were substantially all less than 100 mesh in size.

The spray dried beads were fed to a tumbling zone, where they were agitated in such manner that a "Curtain" or thin falling stream of particles was sprayed with droplets of silicate solution. The silicate employed was of $\text{Na}_2\text{O}/\text{SiO}_2$ ratio of 0.43 or 1 : 2.35, silicates of $\text{Na}_2\text{O}/\text{SiO}_2$ ratios greater than 1 : 2 and less than 1 : 3 being less satisfactory. The detergent beads were sprayed with 15%, 20% and 27% of a 43.5% solution of soluble sodium silicate. The concentration of silicate used may be varied from 25 to 46% and still the treated beads obtained will be satisfactory. However, when the lower concentrations are employed, as when bead moisture is initially low, it is usually desirable to increase the amount of solution applied to assure that enough silicate is on the bead surfaces. Increased moisture content in the silicate solution tends to cause greater agglomeration of beads so this must be kept in mind when using more dilute silicates.

After spraying with silicate the detergent particles were tumbled for about 15 minutes so that the silicate was evenly distributed. During tumbling some finer particles were agglomerated, product density was decreased and particle size was raised to 6 to 60 mesh.

The silicate coated beads, their moisture content raised to about 16%, 20% and 24% respectively, were immediately compacted at a pressure of about 70 pounds per square inch, to form a shape-retaining briquette, held together by the silicate coating to a substantial extent but not so tightly compacted as objectionably to resist disintegration in water under washing conditions. The briquettes made were sprayed with polyvinyl alcohol of weight average molecular weight of about 100 and polyvinyl acetate content of about 20%, 0.8 to 1% of the technical polyvinyl alcohol was applied evenly to the briquette by spraying as an aqueous solution and the coating, an irregular film of an area of about 15 square inches, was heat dried to hardness.

In place of the alkyl benzene sulphonate in this formula one may use other anionic detergents such as sodium lauryl sulphate or N-higher fatty acyl-N-methyl tauride. The non-ionic detergent may be partially replaced by other non-ionics such as the block copolymers of ethylene oxide and propylene oxide (Pluronic F-68) and polyethoxy esters of higher fatty acids and alcohols. Also the fatty alcohol may sometimes be replaced with fatty acids of about the same chain length.

The finished tablets were attractive, non-dusting and sufficiently strong to be handled in commerce without breakage. Of course, they should be packed so that there is no

excessive shaking or rattling in shipment. Also, a moisture barrier packing will keep the tablets from drying out to a more brittle state. These tablets withstood drop tests and simulated shipping tests. Even though they are so strong, the tablets still disintegrated rapidly in water in a washing machine. No undissolved lumps were found in clothing, even after washing with warm or cool water. Usually freshly made briquettes dissolved within about 60 seconds in water at 100°F. agitated to simulate normal washing motions.

Example II

Ethenoxylated nonyl phenol (9.5 ethylene oxide)	13	80
Sodium tridecyl benzene sulphonate	5		
Technical cetyl alcohol	1	
Sodium tripolyphosphate	43	
Sodium sulphate	25	85
Sodium silicate ($\text{Na}_2\text{O}/\text{SiO}_2$ ratio of 1 : 2.35)	4	
Moisture	8.5	
Adjuvants (anti-redeposition agent, fluorescent brightener, perfume, antioxidant)	0.5	90
			100.0

The above formula was made by spray drying an aqueous crutcher mix slurry of all materials except perfume, which was added after spraying. In a tumbling drum there were added to 100-X parts of spray dried particles of such formula of size from 8 to 100 mesh by spraying the following amounts and concentrations of a water solution of sodium silicate of $\text{Na}_2\text{O}/\text{SiO}_2$ ratio of 1 : 2.35. Moisture contents of briquetted tablets made therefrom are also given.

(X) Parts Silicate Solution	Percentage Solids	Final Moisture	105
20	43.5	18.6	
23.7	43.5	20.0	
19.8	39.0	17.0	
24.7	39.0	20.2	
19.0	37.0	19.0	110
20.3	37.0	20.0	
26.3	34.7	23.6	
18.8	34.7	20.7	

After about 5 minutes tumbling, talc powder, substantially all less than 200 mesh, was air-blown onto the tumbling particles. The amount of talc used was 2% and the beads were agitated for another ten minutes, after which they were of 6 to 60 mesh particle size and were ready for immediate pressing. (The addition of talc is not essential and in comparable experiments satisfactory products have been obtained by practicable methods like that of this example

without talc or other insoluble silicate.) Within a period not greater than two hours, the coated particles were pressed to flat cylinder shape at a pressure within the 40 to 5 90 pounds per square inch range and spray coated with about 0.8% polyvinyl alcohol in a 17% aqueous solution at about 150°F. The polyvinyl alcohol was of weight average 10 degree of polymerization of about 100 and contained 15 to 25% polyvinyl acetate. After 15 coating of the tablet the polyvinyl alcohol was dried under infra-red lamps. The coating made was of irregular thickness, between 0.01 and 0.8 millimetre.

15 The resulting tablets pressed very easily 20 in the automatic machinery employed (having Teflon dies and sleeves) and satisfactorily passed drop tests, shipping tests and disintegration tests (in water). They were 25 attractive in appearance, dust-free and easy to use. The polyvinyl alcohol of the coating also increased the anti-redeposition properties of the detergent. In storage the tablets did not swell or crumble, but retained their original shapes and surface appearances. They could be shipped, even in cold weather, and when packed properly exhibited a surprisingly good resistance to breakage.

WHAT WE CLAIM IS:—

- 30 1. A process of manufacturing a briquetted detergent tablet comprising mixing together water-soluble non-ionic organic detergent and a normally solid water-soluble inorganic salt, producing therefrom a particulate detergent containing up to 21% moisture, applying an aqueous solution of water-soluble silicate to the particulate detergent to coat the particles with 3 to 20% soluble silicate and to increase the moisture 35 content thereof to 16 to 25%, pressing the detergent particles into a solid form-retaining briquetted tablet at a pressure low enough to form a tablet which is readily disintegrable in water and applying to the 40 tablet 0.25 to 5% of a readily water-soluble 45 organic film-forming polymer to form on the briquette surface a water-soluble film which is of strength sufficient to help make the detergent tablet resistant to abrasion and accidental breakage, when dry, and of solubility such that the detergent tablet is readily 50 disintegrable in water.
- 55 2. A process as claimed in claim 1 which comprises mixing together 4 to 14% of a water-soluble alkyl phenoxy poly lower alkenoxy lower alkanol detergent and 30 to 60 75% of a water-soluble inorganic builder salt, producing therefrom a particulate detergent containing 2 to 18% moisture, applying 15 to 30% of an aqueous solution of 65 25 to 46% solids content sodium silicate of $\text{Na}_2\text{O}/\text{SiO}_2$ ratio of 1 : 2 to 1 : 3 to the particulate detergent to coat the particles with 3 to 15% sodium silicate and to increase the moisture content thereof to 17 to 24%.

pressing the detergent particles into a solid form-retaining briquetted tablet at a pressure which is in the range 10 to 100 pounds per square inch and which is low enough to form a tablet which is readily disintegrable in water, and applying to the tablet 0.25 to 5% of a readily water-soluble polyvinyl alcohol.

3 A process as claimed in claim 1 or 70 claim 2 which comprises mixing together an aqueous slurry of 7 to 12% of a water-soluble nonyl phenoxy polyethenoxy ethanol detergent of 9 to 10 ethenoxy groups, 1.0 to 5% of sodium alkyl benzene sulphonate

75 in which the alkyl group is of 12 to 15 carbon atoms, 0.5 to 3% of fatty alcohol foam stabilizer having a fatty alkyl group of 12 to 18 carbon atoms and 30 to 50% of sodium tripolyphosphate builder salt, spray drying the slurry to form particles of moisture content of 7 to 14% and of particle size substantially all between 6 and 100 mesh, tumbling the partially dried particles and, while tumbling, spraying onto the particles 15 to

80 30% of an aqueous solution of 35 to 43.5% solids content sodium silicate of $\text{Na}_2\text{O}/\text{SiO}_2$ ratio of approximately 1 : 2.35 to coat the particles with 6.5 to 8.5% sodium silicate, to increase the moisture content thereof to

85 17.5 to 20.5% and to cause agglomeration into larger particles substantially all within the size range of 6 to 60 mesh, pressing the detergent particles into a solid form-retaining briquetted tablet at a pressure which is in the range 40 to 90 pounds per square inch

90 and which is low enough to form a tablet which is readily disintegrable in water, spraying the tablet with 0.25 to 1.0% readily water-soluble polyvinyl alcohol in aqueous solution, the polyvinyl alcohol having a 95 weight average degree of polymerization of 30 to 300 and a polyvinyl acetate content of about 10 to 30% thereof, and drying the polyvinyl alcohol solution to form a water-

100 soluble protective film coating.

4. A process as claimed in any of the preceding claims in which 0.5 to 5% of a finely divided water-insoluble inorganic sili-

105 cate is applied to the detergent particles after they have been coated with the soluble sili-

110 cate and before pressing them into tablet form.

5. A process as claimed in claim 4 in which the finely divided water-insoluble in-

115 organic silicate applied to the detergent par-

ticles is talc and is applied in an amount of 0.5 to 5%.

6. A process as claimed in any of the preceding claims in which, after application 120 of the film-forming polymer, the briquetted tablets are packed in substantially moisture-

125 tight containers which are then closed.

7. A process of manufacturing a briquetted detergent tablet, substantially as described in either of the Examples.

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8. A briquetted detergent tablet manufactured by a process as claimed in any of the preceding claims.
9. A water-soluble detergent briquette which is resistant to abrasion and accidental breakage, when dry, and disintegrates readily in water, comprising a multiplicity of solid particles composed of a homogeneous mixture of water-soluble non-ionic organic detergent and water-soluble inorganic builder salt, of 16 to 25% moisture content, in the form of adhering particles coated with 3 to 20% water-soluble silicate, the coated particles being lightly compacted and fused together to form a shaped briquette, which is coated with an adhering and binding film of 0.25 to 5% of a readily water-soluble organic film-forming polymer.
10. A detergent briquette as claimed in Claim 9 in which the readily water-soluble synthetic organic film-forming polymer is a polyvinyl alcohol of a weight average degree of polymerization of 30 to 300 and a polyvinyl acetate content of 10 to 30% and the coating thereof has an average thickness of 0.01 to 0.8 millimetre.
11. A detergent briquette as claimed in Claim 9 or Claim 10 in which the solid detergent particles are spray dried particles and comprise a mixture of 7 to 12% of nonyl phenoxy polyethenoxy ethanol of 9 to 10 ethenoxy groups, and 30 to 50% of sodium tripolyphosphate, the particles are coated with 6.5 to 8.5% of sodium silicate of Na₂O/SiO₂ ratio of 1 : 2.35, the coated particles are in the form of adhering particles of moisture content of 17.5 to 20.5% and of diameters substantially within the range of 0.25 to 4 millimetres, and the compacted tablet is coated with 0.25 to 1% of a polyvinyl alcohol of a weight average degree of polymerization of approximately 100 and a polyvinyl acetate content of 15 to 25%, which forms on the surface of substantially the whole detergent briquette a water-soluble coating of thickness averaging 0.02 to 0.1 millimetre.
12. A detergent briquette as claimed in any of claims 9 to 11 in which 0.5 to 5% of finely divided water-insoluble silicate coats the particles over the soluble silicate coating.
13. A detergent briquette as claimed in any of claims 9 to 12 packed in a substantially moisture-tight container.

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